Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Di-μ-acetato-κ^4O:O'-tetracarbonylbis(pyridyl-2-yl-κN-amine)diruthenium(I)(*Ru—Ru*)

Reto Dorta,‡ Georg Süss-Fink and Helen Stoeckli-Evans*

Institut de Chimie, Université de Neuchâtel, Avenue de Bellevaux 51, CH-2000 Neuchâtel, Switzerland

‡ Present address: The Arnold and Mabel Beckman Laboratories of Chemical Synthesis, California Institute of Technology, Pasadena, California 91125, USA.

Correspondence e-mail: helen.stoeckli-evans@unine.ch

Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.005 Å
R factor = 0.025
wR factor = 0.058
Data-to-parameter ratio = 13.4

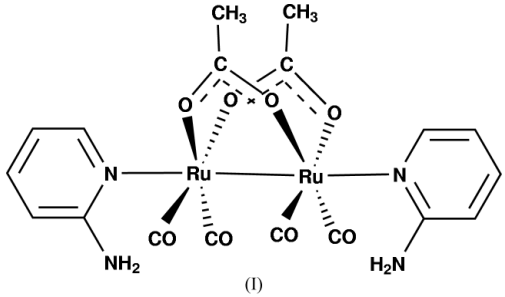
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 26 August 2004
Accepted 2 September 2004
Online 11 September 2004

The structure of the title compound, $[Ru_2(C_2H_3O_2)_2(C_5H_6N_2)_2(CO)_4]$, consists of a 'sawhorse' diruthenium complex with two acetate bridges and two 2-aminopyridine ligands in the axial positions, coordinating *via* the pyridine N atoms. A double-stranded hydrogen-bonded chain is formed *via* intra- and intermolecular hydrogen bonds involving the amine groups, one O atom of both acetate groups and two carbonyl O atoms.

Comment

Binuclear ruthenium(I) 'sawhorse' complexes of the type $[Ru_2(CO)_4(\mu_2,\eta^2\text{-}OOCR)_2L_2]$ were first discovered by Crooks *et al.* (1969). These complexes were prepared by refluxing $[Ru_3(CO)_{12}]$ with the corresponding acid (RCO_2H), which led first to the formation of the polymer $[Ru_2(CO)_4(\mu_2,\eta^2\text{-}OOCR)_2]_n$. The latter was then dissolved in a coordinating solvent (L), such as tetrahydrofuran (THF) or CH_3CN, to give the binuclear complex $[Ru_2(CO)_4(\mu_2,\eta^2\text{-}OOCR)_2(L)_2]$. It was shown later by Rheinwald (1994) that complexes of the type $[Ru_2(CO)_4(\mu_2,\eta^2\text{-}OOCR)_2(THF)_2]$ could be synthesized directly, and almost quantitatively, by the reaction of $[Ru_3(CO)_{12}]$ with the corresponding acid in THF at 393 K by using a pressure Schlenk tube. Reaction of the bisacetate derivative with 2-aminopyridine in THF led to the formation of the title compound, (I).



(I)

The molecular structure of (I) is shown in Fig. 1 and selected geometric parameters are given in Table 1. In this binuclear ruthenium(I) 'sawhorse' complex, the two aminopyridine molecules occupy the axial positions and are coordinated *via* the pyridine N atoms. The pyridine rings do not lie in the same plane but are inclined with respect to one another by 75.45 (17)°. Amine atom N4 is intramolecularly hydrogen bonded to acetate atom O4 (details are given in Fig. 2 and Table 2). The pyridine rings are displaced away from the carbonyl groups with N1—Ru1—Ru2 and N3—Ru2—Ru1 angles of 160.31 (7) and 164.55 (7)°, respectively. This arrangement was also seen in the pyridine analogue (Kepert *et*

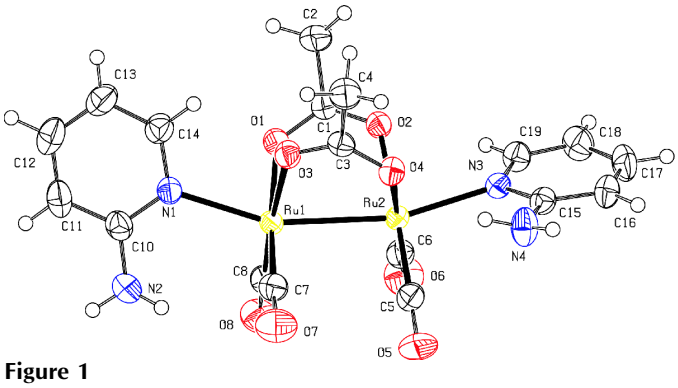


Figure 1
A perspective view of (I), showing 50% probability displacement ellipsoids and the atom-numbering scheme.

al., 2000) and the di(2-pyridyl)amine analogue (Kepert *et al.*, 2003), both of which possess C_2 symmetry.

The overall geometry of the diruthenium(I) 'sawhorse' moiety is also similar to that in the above-mentioned complexes. The Ru1—Ru2 distance is 2.6756 (7) Å, and the Ru1—N1 and Ru2—N3 distances are 2.267 (3) and 2.243 (3) Å, respectively. The Ru—N1 distance is slightly longer than that observed previously. The acetate groups are slightly twisted with respect to the Ru1—Ru2 bond (by *ca* 15°), as shown by the O1—Ru1—Ru2—O2 and O3—Ru1—Ru2—O4 torsion angles (Table 1).

Amine atom N2 is intermolecularly hydrogen bonded to carbonyl atoms O8^{i} and O5^{ii} (see Table 2 for symmetry codes and details). In this way, a polymer chain is built up, as illustrated in Fig. 2. The aminopyridine ligand involving amine atom N4 is also involved in an intermolecular hydrogen bond to acetate atom O1^{iii}. This results in the formation of a double-stranded hydrogen-bonded chain, extending in the *a* direction. This situation is illustrated in Fig. 3 and full details of the hydrogen bonding are given in Table 2.

Experimental

Using a pressure Schlenk tube, 2-aminopyridine (67 mg, 0.712 mmol) was added, under argon and with stirring, to an orange solution of $[Ru_2(CO)_4(\mu_2,\eta^2\text{-}OOCCH_3)_2(THF)_2]$ (200 mg, 0.346 mmol) in THF (50 ml). The solution became yellow and was stirred at room temperature for 30 min. The solvent was evacuated under vacuum, and the resulting residue was redissolved in CH_2Cl_2 (10 ml) and then purified by thin-layer chromatography (CH_2Cl_2/cyclohexane 2:1). The yellow band, containing the title compound, (I), was extracted with CH_2Cl_2 and dried *in vacuo*, giving a yellow crystalline solid. Yield 33 mg (86%). The elemental and spectroscopic analyses agreed with the structure deduced by X-ray crystallography.

Crystal data

$[Ru_2(C_2H_3O_2)_2(C_5H_6N_2)_2(CO)_4]$	$Z = 2$
$M_r = 620.50$	$D_x = 1.871$ Mg m^{-3}
Triclinic, $P\overline{1}$	Mo $K\alpha$ radiation
$a = 9.3027$ (15) Å	Cell parameters from 23 reflections
$b = 9.952$ (2) Å	$\theta = 14.0$–$16.8°$
$c = 12.474$ (3) Å	$\mu = 1.42$ mm^{-1}
$\alpha = 83.82$ (2)°	$T = 293$ (2) K
$\beta = 78.925$ (17)°	Block, yellow
$\gamma = 76.881$ (15)°	0.34 × 0.34 × 0.19 mm
$V = 1101.3$ (4) Å^3	

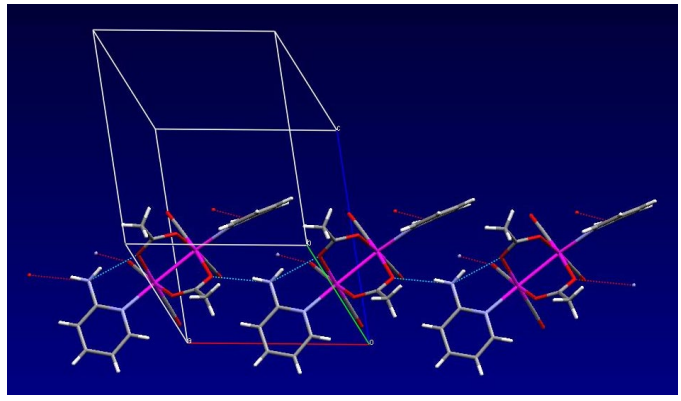


Figure 2
A view of part of the crystal packing of (I), showing the formation of the hydrogen-bonded chain along the *a* axis (hydrogen bonds are represented by dashed blue lines).

Data collection

Stoe AED-2 four-circle diffractometer	3678 reflections with $I > 2\sigma(I)$
$2\theta/\omega$ scans	$\theta_{max} = 25.5°$
Absorption correction: refined from ΔF (DELrefABS in *PLATON*; Spek, 2003)	$h = -10 \rightarrow 11$
$T_{min} = 0.363$, $T_{max} = 0.764$	$k = -11 \rightarrow 12$
4092 measured reflections	$l = 0 \rightarrow 15$
4092 independent reflections	3 standard reflections
	frequency: 60 min
	intensity decay: 1%

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0207P)^2 + 1.3224P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)] = 0.025$	$(\Delta/\sigma)_{max} = 0.001$
$wR(F^2) = 0.058$	$\Delta\rho_{max} = 0.42$ e Å^{-3}
$S = 1.12$	$\Delta\rho_{min} = -0.38$ e Å^{-3}
4092 reflections	
305 parameters	
H atoms treated by a mixture of independent and constrained refinement	

Table 1
Selected geometric parameters (Å, °).

Ru1—C8	1.833 (3)	Ru2—C6	1.830 (3)
Ru1—C7	1.834 (4)	Ru2—C5	1.837 (4)
Ru1—O1	2.131 (2)	Ru2—O2	2.127 (2)
Ru1—O3	2.132 (2)	Ru2—O4	2.133 (2)
Ru1—N1	2.267 (3)	Ru2—N3	2.243 (3)
Ru1—Ru2	2.6756 (7)		
C8—Ru1—C7	90.59 (15)	C6—Ru2—C5	88.00 (16)
C8—Ru1—O1	95.02 (12)	C6—Ru2—O2	94.91 (13)
C7—Ru1—O1	173.35 (12)	C5—Ru2—O2	176.54 (13)
C8—Ru1—O3	175.45 (12)	C6—Ru2—O4	176.91 (12)
C7—Ru1—O3	91.21 (12)	C5—Ru2—O4	95.01 (13)
O1—Ru1—O3	82.94 (9)	O2—Ru2—O4	82.06 (9)
C8—Ru1—N1	98.68 (12)	C6—Ru2—N3	93.14 (13)
C7—Ru1—N1	102.03 (13)	C5—Ru2—N3	98.45 (13)
O1—Ru1—N1	80.68 (9)	O2—Ru2—N3	83.27 (9)
O3—Ru1—N1	85.03 (9)	O4—Ru2—N3	87.11 (9)
C8—Ru1—Ru2	93.73 (10)	C6—Ru2—Ru1	94.75 (11)
C7—Ru1—Ru2	93.02 (11)	C5—Ru2—Ru1	95.08 (11)
O1—Ru1—Ru2	83.07 (6)	O2—Ru2—Ru1	82.83 (6)
O3—Ru1—Ru2	82.01 (6)	O4—Ru2—Ru1	84.31 (6)
N1—Ru1—Ru2	160.31 (7)	N3—Ru2—Ru1	164.55 (7)
O1—Ru1—Ru2—O2	16.05 (9)	O3—Ru1—Ru2—O4	14.93 (9)

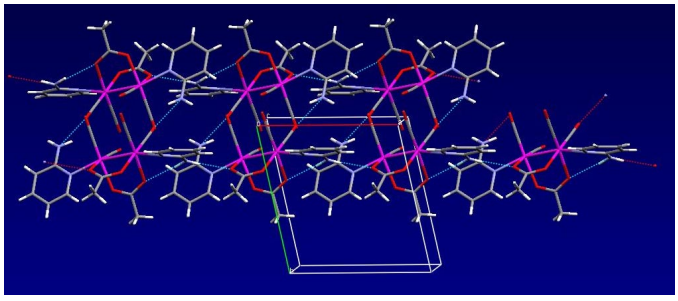


Figure 3
A view of the formation of the double-stranded hydrogen-bonded chain in (I), extending in the *a* direction (hydrogen bonds are represented by dashed blue lines).

Table 2
Hydrogen-bonding geometry (Å, °).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H21···O8^{i}	0.880 (19)	2.60 (2)	3.461 (4)	166 (4)
N2—H22···O5^{ii}	0.865 (19)	2.39 (3)	3.031 (5)	132 (3)
N4—H41···O1^{iii}	0.874 (18)	2.17 (2)	3.032 (4)	171 (3)
N4—H42···O4	0.882 (19)	2.24 (3)	3.050 (4)	152 (3)

Symmetry codes: (i) $1-x, -y, 1-z$; (ii) $2-x, -y, 1-z$; (iii) $1+x, y, z$.

The amine H atoms were located in a difference map and were refined isotropically, with the N—H distance restrained to 0.88 (2) Å. The remaining H atoms were included in calculated positions and treated as riding atoms, with C—H = 0.96 Å and $U_{iso}(H) = 1.5U_{eq}(C)$ for CH_3 atoms, and C—H = 0.93 Å and $U_{iso}(H) = 1.2U_{eq}(C)$ for aromatic H atoms.

Data collection: *STADI*4 (Stoe & Cie, 1997); cell refinement: *STADI*4; data reduction: *X-RED* (Stoe & Cie, 1997); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1990); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003) and *MERCURY* (Bruno *et al.*, 2002); software used to prepare material for publication: *SHELXL*97.

This work was supported by the Swiss National Science Foundation.

References

Bruno, I. J., Cole, J. C., Edgington, P. R., Kessler, K., Macrae, C. F., McCabe, P., Pearson, J. & Taylor, R. (2002). *Acta Cryst.* B**58**, 389–397.
Crooks, G. R., Johnson, B. F. G., Lewis, J., Williams, I. G. & Gamlen, G. (1969). *J. Chem. Soc. A*, pp. 2761–2766.
Kepert, C. M., Deacon, G. B., Spiccia, L., Fallon, G. D. & Skelton, B. W. (2000). *J. Chem. Soc. Dalton Trans.* pp. 2867–2873.
Kepert, C. M., Deacon, G. B. & Spiccia, L. (2003). *Inorg. Chim. Acta*, **355**, 213–222.
Rheinwald, G. (1994). Doctoral thesis, University of Neuchâtel, Switzerland.
Sheldrick, G. M. (1990). *Acta Cryst.* A**46**, 467–473.
Sheldrick, G. M. (1997). *SHELXL*97. University of Gottingen, Germany.
Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.
Stoe & Cie (1997). *STADI*4 and *X-RED*. Stoe & Cie, Darmstadt, Germany.

supporting information

Acta Cryst. (2004). E**60**, m1397–m1399 [doi:10.1107/S160053680402166X]

Di-μ-acetato-$\kappa^4O:O'$-tetracarbonylbis(pyridyl-2-yl-κN-amine)diruthenium(I)(*Ru—Ru*)

Reto Dorta, Georg Süss-Fink and Helen Stoeckli-Evans

S1. Comment

Binuclear ruthenium(I) `sawhorse′ complexes of the type $[Ru_2(CO)_4(\mu_2,\eta^2\text{-}OOCR)_2L_2]$ were first discovered by Crooks *et al.* (1969). These complexes were prepared by refluxing $[Ru_3(CO)_{12}]$ with the corresponding acid (RCO_2H), which lead first to the formation of the polymer $[Ru_2(CO)_4(\mu_2,\eta^2\text{-}OOCR)_2]_n$. The latter was then dissolved in a coordinating solvent (*L*), such as tetrahydrofuran (THF) or CH_3CN, to give the binuclear complex $[Ru_2(CO)_4(\mu_2,\eta^2\text{-}OOCR)_2(L)_2]$. It was shown later by Rheinwald (1994) that complexes of the type $[Ru_2(CO)_4(\mu_2,\eta^2\text{-}OOCR)_2(THF)_2]$ could be synthesized directly and almost quantitatively by the reaction of $[Ru_3(CO)_{12}]$ with the corresponding acid in THF at 393 K by using a pressure Schlenk tube. Reaction of the bisacetate derivative with 2-aminopyridine in THF lead to the formation of the title compound, (I).

The molecular structure of (I) is shown in Fig. 1 and selected geometric parameters are given in Table 1. In this binuclear ruthenium(I) `sawhorse′ complex, the two aminopryidine molecules occupy the axial positions and are coordinated *via* the pyridine N atoms. The pyridine rings do not lie in the same plane but are inclined with respect to one another by 75.45 (17)°. Amine atom N4 is intramolecularly hydrogen bonded to acetate atom O4 (details are given in Fig. 2 and Table 2). The pyridine rings are displaced away from the carbonyl groups with N1—Ru1—Ru2 and N3—Ru2—Ru1 angles of 160.31 (7) and 164.55 (7)°, respectively. This arrangement was also seen in the pyridine analogue (Kepert *et al.*, 2000) and the di(2-pyridyl)amine analogue (Kepert *et al.*, 2003), both of which possess C_2 symmetry.

The overall geometry of the diruthenium(I) `sawhorse′ moiety is also similar to that in the above-mentioned complexes. The Ru1···Ru2 distance is 2.6756 (7) Å, and the Ru1—N1 and Ru2—N3 distances are 2.267 (3) and 2.243 (3) Å, respectively. The Ru—N1 distance is slightly longer than that observed previously. The acetate groups are slightly twisted with respect to the Ru1—Ru2 bond (by *ca* 15°), as shown by the O1—Ru1—Ru2—O2 and O3—Ru1—Ru2—O4 torsion angles (Table 1).

Amine atom N2 is intermolecularly hydrogen bonded to carbonyl atoms $O8^{i}$ and $O5^{ii}$ (see Table 2 for symmetry codes and details). In this way, a polymer chain is built up, as illustrated in Fig. 2. The aminopyridine ligand involving amine atom N4 is also involved in an intermolecular hydrogen bond to acetate atom $O1^{iii}$. This results in the formation of a double-stranded hydrogen-bonded chain, extending in the a direction. This situtaion is illustrated in Fig. 3 and full details of the hydrogen bonding are given in Table 2.

S2. Experimental

Using a pressure Schlenk tube, 2-aminopyridine (67 mg, 0.712 mmol) was added, under argon and with stirring, to an orange solution of $[Ru_2(CO)_4(\mu_2,\eta^2\text{-}OOCCH_3)_2(THF)_2]$ (200 mg, 0.346 mmol) in THF (50 ml). The solution became yellow and was stirred at room temperature for 30 min. The solvent was evacuated under vacuum, and the resulting residue was redissolved in CH_2Cl_2(10 ml) and then purified by thin-layer chromatography (CH_2Cl_2/cyclohexane 2:1). The

yellow band, containing the title compound, (I), was extracted with CH_2Cl_2 and dried *in vacuo*, giving a yellow crystalline solid. Yield 33 mg (86%). The elemental and spectrocopic analyses agreed with the structure deduced by X-ray crystallography.

S3. Refinement

The amine H atoms were located from a difference map and were refined isotropically, with the N—H distance restrained to 0.88 (2) Å. The remaining H atoms were included in calculated positions and treated as riding atoms using *SHELXL97* (Sheldrick, 1997) default parameters [C—H = 0.96 Å and U_{iso}(H) = 1.5U_{eq}(C) for CH_3 atoms, and C—H = 0.93 Å and U_{iso}(H) = 1.2U_{eq}(C) for aromatic H atoms].

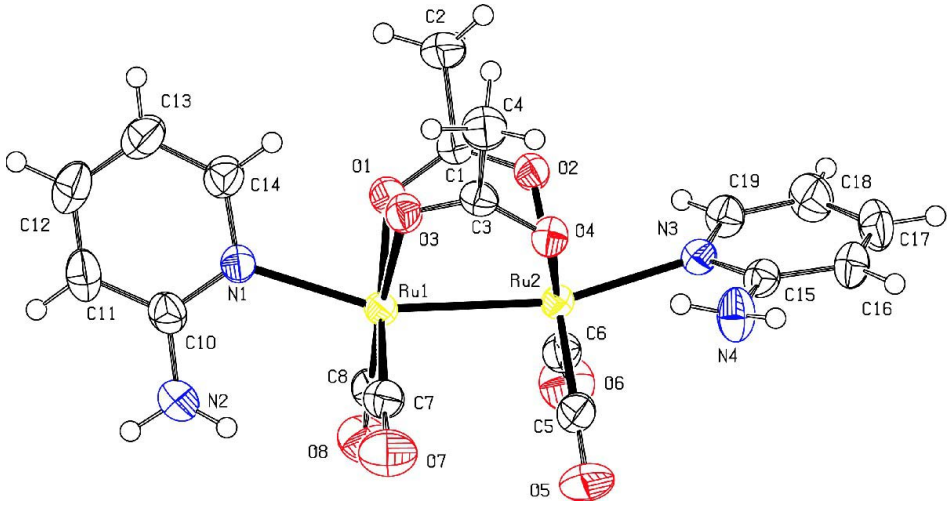


Figure 1

A perspective view of (I), showing 50% probability displacement ellipsoids and the atom-numbering scheme.

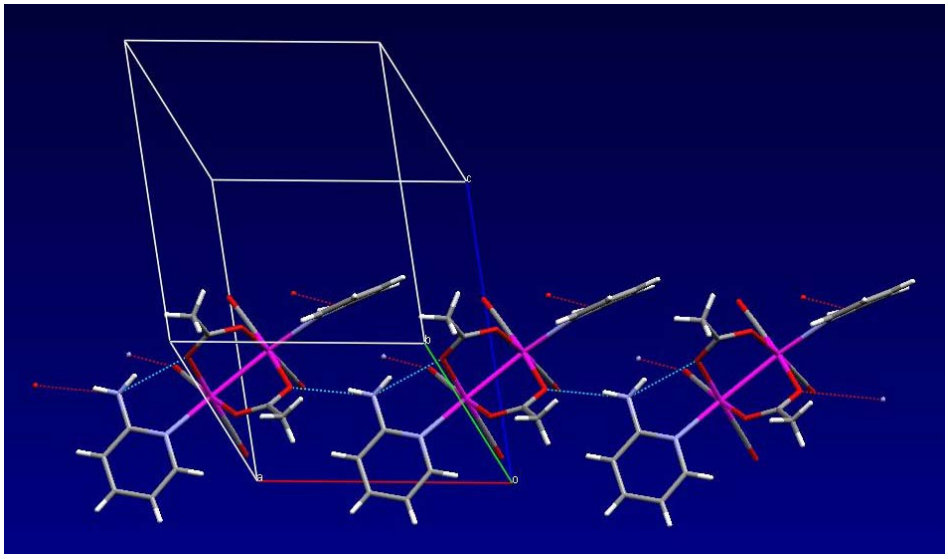


Figure 2

A view of the crystal packing of (I), showing the formation of the hydrogen-bonded chain along the *a* axis (hydrogen bonds are represented by dashed blue lines).

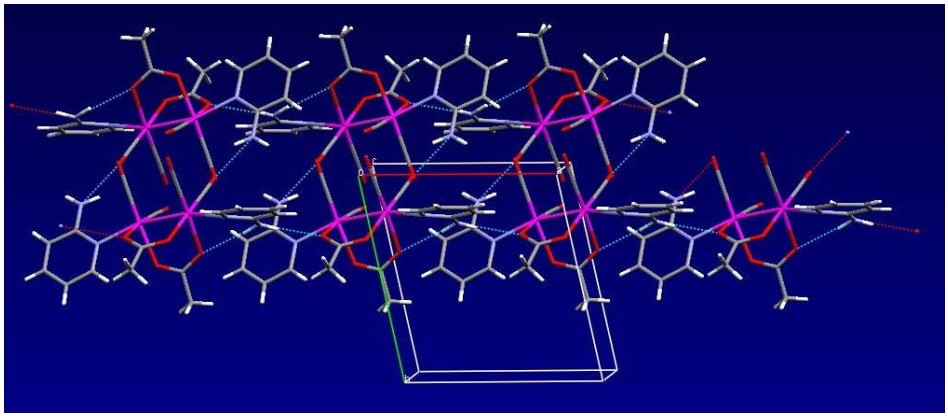


Figure 3

A view of the formation of the double-stranded hydrogen-bonded chain in (I), extending in the a direction (hydrogen bonds are represented by dashed blue lines).

Di-μ-acetato-κ^4O:O'-tetracarbonylbis(pyridyl-2-yl-κN- amine)diruthenium(I)(Ru—Ru)

Crystal data

$[Ru_2(C_2H_3O_2)_2(C_5H_6N_2)_2(CO)_4]$
$M_r = 620.50$
Triclinic, $P\overline{1}$
Hall symbol: -P 1
$a = 9.3027$ (15) Å
$b = 9.952$ (2) Å
$c = 12.474$ (3) Å
$\alpha = 83.82$ (2)°
$\beta = 78.925$ (17)°
$\gamma = 76.881$ (15)°
$V = 1101.3$ (4) Å^3
$Z = 2$
$F(000) = 612$
$D_x = 1.871$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 23 reflections
$\theta = 14.0$–16.8°
$\mu = 1.42$ mm^{-1}
$T = 293$ K
Block, yellow
0.34 × 0.34 × 0.19 mm

Data collection

Stoe AED2 four-circle diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
$2\theta/\omega$ scans
Absorption correction: part of the refinement model (ΔF)
(DELrefABS in *PLATON*; Spek, 2003)
$T_{min} = 0.363$, $T_{max} = 0.764$
4092 measured reflections
4092 independent reflections
3678 reflections with $I > 2\sigma(I)$
$R_{int} = 0.000$
$\theta_{max} = 25.5$°, $\theta_{min} = 2.1$°
$h = -10 \rightarrow 11$
$k = -11 \rightarrow 12$
$l = 0 \rightarrow 15$
3 standard reflections every 60 min
intensity decay: 1%

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.025$
$wR(F^2) = 0.058$
$S = 1.12$
4092 reflections
305 parameters
4 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0207P)^2 + 1.3224P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$

$\Delta\rho_{max} = 0.42$ e Å^{-3}
$\Delta\rho_{min} = -0.38$ e Å^{-3}

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit *S* are based on F^2, conventional *R*-factors *R* are based on *F*, with *F* set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on *F*, and *R*- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
Ru1	0.81649 (3)	0.24089 (2)	0.32290 (2)	0.02656 (7)
Ru2	1.08333 (3)	0.18889 (2)	0.19130 (2)	0.02594 (7)
O1	0.7364 (2)	0.3021 (2)	0.17226 (17)	0.0342 (5)
O2	0.9619 (2)	0.3138 (2)	0.07518 (17)	0.0329 (5)
O3	0.8575 (2)	0.4451 (2)	0.30466 (18)	0.0344 (5)
O4	1.1011 (2)	0.3856 (2)	0.23392 (18)	0.0333 (5)
O5	1.2456 (3)	0.0260 (3)	0.3636 (3)	0.0683 (9)
O6	1.0494 (3)	−0.0786 (3)	0.1224 (3)	0.0630 (8)
O7	0.9554 (3)	0.1836 (3)	0.5242 (2)	0.0626 (8)
O8	0.7785 (3)	−0.0503 (3)	0.3327 (2)	0.0564 (7)
N1	0.5768 (3)	0.3439 (3)	0.3892 (2)	0.0330 (6)
N2	0.5285 (4)	0.1594 (4)	0.5101 (3)	0.0593 (10)
H21	0.462 (4)	0.125 (5)	0.558 (3)	0.075 (15)*
H22	0.623 (2)	0.126 (4)	0.509 (3)	0.055 (12)*
N3	1.2812 (3)	0.1995 (3)	0.0582 (2)	0.0300 (6)
N4	1.4300 (3)	0.2434 (4)	0.1744 (3)	0.0457 (8)
H41	1.513 (3)	0.266 (3)	0.180 (3)	0.027 (8)*
H42	1.346 (3)	0.286 (4)	0.214 (3)	0.052 (12)*
C1	0.8211 (3)	0.3464 (3)	0.0908 (3)	0.0297 (7)
C2	0.7477 (4)	0.4465 (4)	0.0084 (3)	0.0502 (10)
H2C	0.8104	0.5106	−0.0218	0.075*
H2B	0.6523	0.4961	0.0434	0.075*
H2A	0.7335	0.3968	−0.0493	0.075*
C3	0.9837 (4)	0.4731 (3)	0.2668 (2)	0.0304 (7)
C4	0.9936 (4)	0.6223 (4)	0.2581 (3)	0.0465 (9)
H4A	0.9275	0.6680	0.3185	0.070*
H4B	0.9649	0.6658	0.1906	0.070*
H4C	1.0946	0.6288	0.2595	0.070*
C5	1.1840 (4)	0.0895 (4)	0.2972 (3)	0.0399 (8)
C6	1.0606 (4)	0.0249 (3)	0.1502 (3)	0.0378 (8)
C7	0.8998 (4)	0.2024 (4)	0.4475 (3)	0.0379 (8)
C8	0.7921 (4)	0.0624 (4)	0.3286 (3)	0.0352 (7)

C10	0.4779 (4)	0.2826 (4)	0.4584 (3)	0.0390 (8)
C11	0.3250 (4)	0.3427 (4)	0.4776 (3)	0.0497 (10)
H11A	0.2576	0.2959	0.5224	0.060*
C12	0.2755 (4)	0.4707 (4)	0.4301 (3)	0.0517 (10)
H12A	0.1743	0.5128	0.4436	0.062*
C13	0.3765 (4)	0.5370 (4)	0.3618 (3)	0.0489 (9)
H13A	0.3452	0.6245	0.3291	0.059*
C14	0.5236 (4)	0.4711 (4)	0.3435 (3)	0.0412 (8)
H14A	0.5913	0.5160	0.2970	0.049*
C15	1.4171 (3)	0.2103 (3)	0.0748 (3)	0.0332 (7)
C16	1.5427 (4)	0.1841 (4)	−0.0085 (3)	0.0409 (8)
H16A	1.6372	0.1855	0.0053	0.049*
C17	1.5261 (4)	0.1569 (4)	−0.1093 (3)	0.0505 (10)
H17A	1.6091	0.1401	−0.1649	0.061*
C18	1.3843 (4)	0.1540 (4)	−0.1294 (3)	0.0494 (9)
H18A	1.3697	0.1393	−0.1985	0.059*
C19	1.2678 (4)	0.1736 (4)	−0.0435 (3)	0.0377 (8)
H19A	1.1735	0.1690	−0.0555	0.045*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Ru1	0.02682 (13)	0.02788 (14)	0.02494 (13)	−0.00673 (10)	−0.00407 (10)	−0.00038 (10)
Ru2	0.02490 (13)	0.02492 (13)	0.02803 (14)	−0.00448 (9)	−0.00487 (10)	−0.00303 (10)
O1	0.0293 (11)	0.0447 (13)	0.0294 (12)	−0.0091 (10)	−0.0074 (9)	0.0013 (10)
O2	0.0301 (11)	0.0383 (12)	0.0293 (12)	−0.0069 (9)	−0.0041 (9)	−0.0003 (10)
O3	0.0332 (12)	0.0297 (12)	0.0388 (13)	−0.0067 (9)	−0.0013 (10)	−0.0047 (10)
O4	0.0315 (11)	0.0315 (12)	0.0377 (13)	−0.0077 (9)	−0.0041 (10)	−0.0071 (10)
O5	0.0615 (19)	0.074 (2)	0.0622 (19)	−0.0006 (16)	−0.0262 (16)	0.0256 (16)
O6	0.077 (2)	0.0413 (16)	0.078 (2)	−0.0230 (14)	−0.0076 (16)	−0.0192 (15)
O7	0.075 (2)	0.076 (2)	0.0434 (16)	−0.0142 (16)	−0.0308 (15)	0.0017 (14)
O8	0.0684 (19)	0.0389 (15)	0.0655 (19)	−0.0219 (13)	−0.0082 (15)	−0.0028 (13)
N1	0.0293 (13)	0.0368 (15)	0.0313 (15)	−0.0062 (11)	−0.0027 (11)	−0.0019 (12)
N2	0.045 (2)	0.061 (2)	0.059 (2)	−0.0081 (18)	0.0034 (18)	0.0204 (18)
N3	0.0297 (13)	0.0302 (14)	0.0302 (14)	−0.0054 (11)	−0.0058 (11)	−0.0031 (11)
N4	0.0318 (16)	0.068 (2)	0.0434 (18)	−0.0168 (15)	−0.0057 (14)	−0.0190 (16)
C1	0.0326 (17)	0.0295 (16)	0.0284 (16)	−0.0065 (13)	−0.0075 (13)	−0.0035 (13)
C2	0.045 (2)	0.054 (2)	0.048 (2)	−0.0058 (18)	−0.0134 (17)	0.0152 (18)
C3	0.0373 (17)	0.0317 (17)	0.0237 (16)	−0.0081 (14)	−0.0066 (13)	−0.0036 (13)
C4	0.051 (2)	0.0308 (18)	0.057 (2)	−0.0112 (16)	−0.0028 (18)	−0.0052 (17)
C5	0.0350 (18)	0.0360 (18)	0.046 (2)	−0.0048 (14)	−0.0042 (16)	−0.0012 (16)
C6	0.0365 (18)	0.0340 (19)	0.042 (2)	−0.0072 (14)	−0.0037 (15)	−0.0038 (15)
C7	0.0407 (18)	0.0364 (19)	0.0363 (19)	−0.0075 (15)	−0.0068 (15)	−0.0022 (15)
C8	0.0339 (17)	0.040 (2)	0.0324 (18)	−0.0100 (14)	−0.0040 (14)	−0.0023 (14)
C10	0.0405 (19)	0.045 (2)	0.0298 (18)	−0.0091 (16)	−0.0018 (14)	−0.0042 (15)
C11	0.0359 (19)	0.070 (3)	0.041 (2)	−0.0129 (18)	0.0046 (16)	−0.0108 (19)
C12	0.036 (2)	0.065 (3)	0.050 (2)	0.0042 (18)	−0.0065 (17)	−0.019 (2)
C13	0.044 (2)	0.046 (2)	0.052 (2)	0.0055 (17)	−0.0106 (18)	−0.0072 (18)

C14	0.0395 (19)	0.0378 (19)	0.044 (2)	−0.0043 (15)	−0.0044 (16)	−0.0060 (16)
C15	0.0309 (16)	0.0297 (16)	0.0388 (19)	−0.0058 (13)	−0.0049 (14)	−0.0053 (14)
C16	0.0285 (17)	0.046 (2)	0.048 (2)	−0.0087 (15)	−0.0017 (15)	−0.0082 (17)
C17	0.042 (2)	0.059 (2)	0.043 (2)	−0.0077 (18)	0.0120 (17)	−0.0123 (19)
C18	0.054 (2)	0.060 (2)	0.0326 (19)	−0.0084 (19)	−0.0032 (17)	−0.0113 (17)
C19	0.0367 (18)	0.0421 (19)	0.0349 (18)	−0.0073 (15)	−0.0062 (14)	−0.0073 (15)

Geometric parameters (Å, º)

Ru1—C8	1.833 (3)	N4—H41	0.874 (18)
Ru1—C7	1.834 (4)	N4—H42	0.882 (19)
Ru1—O1	2.131 (2)	C1—C2	1.502 (4)
Ru1—O3	2.132 (2)	C2—H2C	0.96
Ru1—N1	2.267 (3)	C2—H2B	0.96
Ru1—Ru2	2.6756 (7)	C2—H2A	0.96
Ru2—C6	1.830 (3)	C3—C4	1.499 (4)
Ru2—C5	1.837 (4)	C4—H4A	0.96
Ru2—O2	2.127 (2)	C4—H4B	0.96
Ru2—O4	2.133 (2)	C4—H4C	0.96
Ru2—N3	2.243 (3)	C10—C11	1.397 (5)
O1—C1	1.265 (4)	C11—C12	1.365 (6)
O2—C1	1.258 (4)	C11—H11A	0.93
O3—C3	1.260 (4)	C12—C13	1.377 (6)
O4—C3	1.262 (4)	C12—H12A	0.93
O5—C5	1.148 (4)	C13—C14	1.363 (5)
O6—C6	1.154 (4)	C13—H13A	0.93
O7—C7	1.151 (4)	C14—H14A	0.93
O8—C8	1.152 (4)	C15—C16	1.402 (5)
N1—C10	1.342 (4)	C16—C17	1.359 (5)
N1—C14	1.357 (4)	C16—H16A	0.93
N2—C10	1.351 (5)	C17—C18	1.396 (5)
N2—H21	0.880 (19)	C17—H17A	0.93
N2—H22	0.865 (19)	C18—C19	1.365 (5)
N3—C15	1.349 (4)	C18—H18A	0.93
N3—C19	1.355 (4)	C19—H19A	0.93
N4—C15	1.353 (4)		

C8—Ru1—C7	90.59 (15)	C1—C2—H2C	109.5
C8—Ru1—O1	95.02 (12)	C1—C2—H2B	109.5
C7—Ru1—O1	173.35 (12)	H2C—C2—H2B	109.5
C8—Ru1—O3	175.45 (12)	C1—C2—H2A	109.5
C7—Ru1—O3	91.21 (12)	H2C—C2—H2A	109.5
O1—Ru1—O3	82.94 (9)	H2B—C2—H2A	109.5
C8—Ru1—N1	98.68 (12)	O3—C3—O4	124.9 (3)
C7—Ru1—N1	102.03 (13)	O3—C3—C4	117.2 (3)
O1—Ru1—N1	80.68 (9)	O4—C3—C4	117.9 (3)
O3—Ru1—N1	85.03 (9)	C3—C4—H4A	109.5
C8—Ru1—Ru2	93.73 (10)	C3—C4—H4B	109.5

C7—Ru1—Ru2	93.02 (11)	H4A—C4—H4B	109.5
O1—Ru1—Ru2	83.07 (6)	C3—C4—H4C	109.5
O3—Ru1—Ru2	82.01 (6)	H4A—C4—H4C	109.5
N1—Ru1—Ru2	160.31 (7)	H4B—C4—H4C	109.5
C6—Ru2—C5	88.00 (16)	O5—C5—Ru2	179.0 (3)
C6—Ru2—O2	94.91 (13)	O6—C6—Ru2	178.3 (3)
C5—Ru2—O2	176.54 (13)	O7—C7—Ru1	176.7 (3)
C6—Ru2—O4	176.91 (12)	O8—C8—Ru1	179.1 (3)
C5—Ru2—O4	95.01 (13)	N1—C10—N2	118.7 (3)
O2—Ru2—O4	82.06 (9)	N1—C10—C11	121.6 (3)
C6—Ru2—N3	93.14 (13)	N2—C10—C11	119.6 (3)
C5—Ru2—N3	98.45 (13)	C12—C11—C10	119.4 (4)
O2—Ru2—N3	83.27 (9)	C12—C11—H11A	120.3
O4—Ru2—N3	87.11 (9)	C10—C11—H11A	120.3
C6—Ru2—Ru1	94.75 (11)	C11—C12—C13	119.4 (3)
C5—Ru2—Ru1	95.08 (11)	C11—C12—H12A	120.3
O2—Ru2—Ru1	82.83 (6)	C13—C12—H12A	120.3
O4—Ru2—Ru1	84.31 (6)	C14—C13—C12	118.4 (4)
N3—Ru2—Ru1	164.55 (7)	C14—C13—H13A	120.8
C1—O1—Ru1	119.59 (19)	C12—C13—H13A	120.8
C1—O2—Ru2	122.7 (2)	N1—C14—C13	123.8 (4)
C3—O3—Ru1	123.8 (2)	N1—C14—H14A	118.1
C3—O4—Ru2	119.6 (2)	C13—C14—H14A	118.1
C10—N1—C14	117.1 (3)	N3—C15—N4	118.5 (3)
C10—N1—Ru1	125.3 (2)	N3—C15—C16	120.7 (3)
C14—N1—Ru1	116.9 (2)	N4—C15—C16	120.8 (3)
C10—N2—H21	116 (3)	C17—C16—C15	119.8 (3)
C10—N2—H22	122 (3)	C17—C16—H16A	120.1
H21—N2—H22	120 (4)	C15—C16—H16A	120.1
C15—N3—C19	118.0 (3)	C16—C17—C18	119.9 (3)
C15—N3—Ru2	124.7 (2)	C16—C17—H17A	120.1
C19—N3—Ru2	116.7 (2)	C18—C17—H17A	120.1
C15—N4—H41	117 (2)	C19—C18—C17	117.4 (3)
C15—N4—H42	115 (3)	C19—C18—H18A	121.3
H41—N4—H42	118 (3)	C17—C18—H18A	121.3
O2—C1—O1	124.5 (3)	N3—C19—C18	124.0 (3)
O2—C1—C2	118.0 (3)	N3—C19—H19A	118.0
O1—C1—C2	117.5 (3)	C18—C19—H19A	118.0

C8—Ru1—Ru2—C6	16.29 (15)	C8—Ru1—N1—C14	−142.3 (3)
C7—Ru1—Ru2—C6	107.08 (15)	C7—Ru1—N1—C14	125.2 (3)
O1—Ru1—Ru2—C6	−78.32 (13)	O1—Ru1—N1—C14	−48.6 (2)
O3—Ru1—Ru2—C6	−162.12 (13)	O3—Ru1—N1—C14	35.0 (2)
N1—Ru1—Ru2—C6	−112.8 (2)	Ru2—Ru1—N1—C14	−13.9 (4)
C8—Ru1—Ru2—C5	−72.12 (15)	C6—Ru2—N3—C15	−123.6 (3)
C7—Ru1—Ru2—C5	18.67 (15)	C5—Ru2—N3—C15	−35.1 (3)
O1—Ru1—Ru2—C5	−166.73 (13)	O2—Ru2—N3—C15	141.8 (3)
O3—Ru1—Ru2—C5	109.47 (13)	O4—Ru2—N3—C15	59.5 (2)

N1—Ru1—Ru2—C5	158.7 (2)	Ru1—Ru2—N3—C15	115.8 (3)
C8—Ru1—Ru2—O2	110.66 (12)	C6—Ru2—N3—C19	46.8 (3)
C7—Ru1—Ru2—O2	−158.55 (12)	C5—Ru2—N3—C19	135.2 (2)
O1—Ru1—Ru2—O2	16.05 (9)	O2—Ru2—N3—C19	−47.8 (2)
O3—Ru1—Ru2—O2	−67.75 (9)	O4—Ru2—N3—C19	−130.2 (2)
N1—Ru1—Ru2—O2	−18.5 (2)	Ru1—Ru2—N3—C19	−73.9 (4)
C8—Ru1—Ru2—O4	−166.66 (12)	Ru2—O2—C1—O1	−5.0 (4)
C7—Ru1—Ru2—O4	−75.87 (12)	Ru2—O2—C1—C2	174.0 (2)
O1—Ru1—Ru2—O4	98.72 (9)	Ru1—O1—C1—O2	27.0 (4)
O3—Ru1—Ru2—O4	14.93 (9)	Ru1—O1—C1—C2	−152.0 (2)
N1—Ru1—Ru2—O4	64.2 (2)	Ru1—O3—C3—O4	0.8 (4)
C8—Ru1—Ru2—N3	136.7 (3)	Ru1—O3—C3—C4	179.3 (2)
C7—Ru1—Ru2—N3	−132.5 (3)	Ru2—O4—C3—O3	19.7 (4)
O1—Ru1—Ru2—N3	42.1 (3)	Ru2—O4—C3—C4	−158.7 (2)
O3—Ru1—Ru2—N3	−41.7 (3)	C14—N1—C10—N2	−176.3 (3)
N1—Ru1—Ru2—N3	7.6 (3)	Ru1—N1—C10—N2	13.4 (5)
C8—Ru1—O1—C1	−120.6 (2)	C14—N1—C10—C11	4.0 (5)
O3—Ru1—O1—C1	55.3 (2)	Ru1—N1—C10—C11	−166.4 (3)
N1—Ru1—O1—C1	141.4 (2)	N1—C10—C11—C12	−3.9 (6)
Ru2—Ru1—O1—C1	−27.4 (2)	N2—C10—C11—C12	176.3 (4)
C6—Ru2—O2—C1	81.3 (3)	C10—C11—C12—C13	1.6 (6)
O4—Ru2—O2—C1	−98.1 (2)	C11—C12—C13—C14	0.5 (6)
N3—Ru2—O2—C1	173.9 (2)	C10—N1—C14—C13	−1.8 (5)
Ru1—Ru2—O2—C1	−12.9 (2)	Ru1—N1—C14—C13	169.4 (3)
C7—Ru1—O3—C3	78.3 (3)	C12—C13—C14—N1	−0.4 (6)
O1—Ru1—O3—C3	−98.6 (2)	C19—N3—C15—N4	176.5 (3)
N1—Ru1—O3—C3	−179.8 (2)	Ru2—N3—C15—N4	−13.3 (4)
Ru2—Ru1—O3—C3	−14.6 (2)	C19—N3—C15—C16	−5.2 (5)
C5—Ru2—O4—C3	−117.7 (2)	Ru2—N3—C15—C16	165.0 (2)
O2—Ru2—O4—C3	60.4 (2)	N3—C15—C16—C17	4.5 (5)
N3—Ru2—O4—C3	144.0 (2)	N4—C15—C16—C17	−177.2 (4)
Ru1—Ru2—O4—C3	−23.1 (2)	C15—C16—C17—C18	−0.5 (6)
C8—Ru1—N1—C10	28.1 (3)	C16—C17—C18—C19	−2.7 (6)
C7—Ru1—N1—C10	−64.4 (3)	C15—N3—C19—C18	1.9 (5)
O1—Ru1—N1—C10	121.8 (3)	Ru2—N3—C19—C18	−169.1 (3)
O3—Ru1—N1—C10	−154.6 (3)	C17—C18—C19—N3	2.0 (6)
Ru2—Ru1—N1—C10	156.5 (2)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H21···O8^{i}	0.88 (2)	2.60 (2)	3.461 (4)	166 (4)
N2—H22···O5^{ii}	0.87 (2)	2.39 (3)	3.031 (5)	132 (3)
N4—H41···O1^{iii}	0.87 (2)	2.17 (2)	3.032 (4)	171 (3)
N4—H42···O4	0.88 (2)	2.24 (3)	3.050 (4)	152 (3)

Symmetry codes: (i) −*x*+1, −*y*, −*z*+1; (ii) −*x*+2, −*y*, −*z*+1; (iii) *x*+1, *y*, *z*.